

*Progress Report #1 for:*

***“Concrete Environmental Monitoring Sensor”***



Colorado Street Bridge, 1988, courtesy Library of Congress.

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## ***1. Project Background***

With infrastructure costs escalating it is becoming essential to monitor the health of concrete structures so that timely maintenance can maximize their useful lives. This SBIR will demonstrate the feasibility of using a passive sensor, embedded within concrete, to measure moisture, temperature, pH and concentration of chlorides. It will provide critical data for evaluating concrete performance starting with the initial quality control period of freshly mixed or freshly cast concrete, through its useful service life, to the period of deterioration and repair. Data obtained from these sensors will result in longer service life, lower infrastructure costs and the development of more effective means of remediation. The device will be powered and interrogated using radio frequency energy from a distance of over one meter, returning a unique identification number so that data can be correlated with sensor location.

## ***2. Summary of Project Objective***

### ***2.1 Objectives***

- Develop and test an RFID communications system
- Integrate temperature and humidity monitoring sensors into the RFID system
- Test the performance of the system in measuring properties of concrete

### ***2.2 Questions to Answer***

- What are the best ways to passively measure temperature, humidity, pH, Cl ion concentration?
- How do we need to package the sensors so that they are protected from the environment but are still able to measure?
- What is the maximum range for the RF signal in concrete and how can we improve it?

## ***3. Tasks Performed for Progress Report #1***

### ***3.1 Literature and Product Search***

The preliminary work included collecting background information on properties of concrete and the parameters that influence the performance of concrete in structural applications. The literature search found several research publications on factors affecting concrete strength and measurement techniques in cement and concrete. Patents on innovative new concrete monitoring methods and new types of sensors were studied. A product search was also done for commercially available products that can be used for concrete monitoring and sensing methods that they employ.

The goal for the final device is to be able to monitor temperature, moisture content, pH, and chloride ion concentration. For temperature monitoring, there is already a wide range of com-

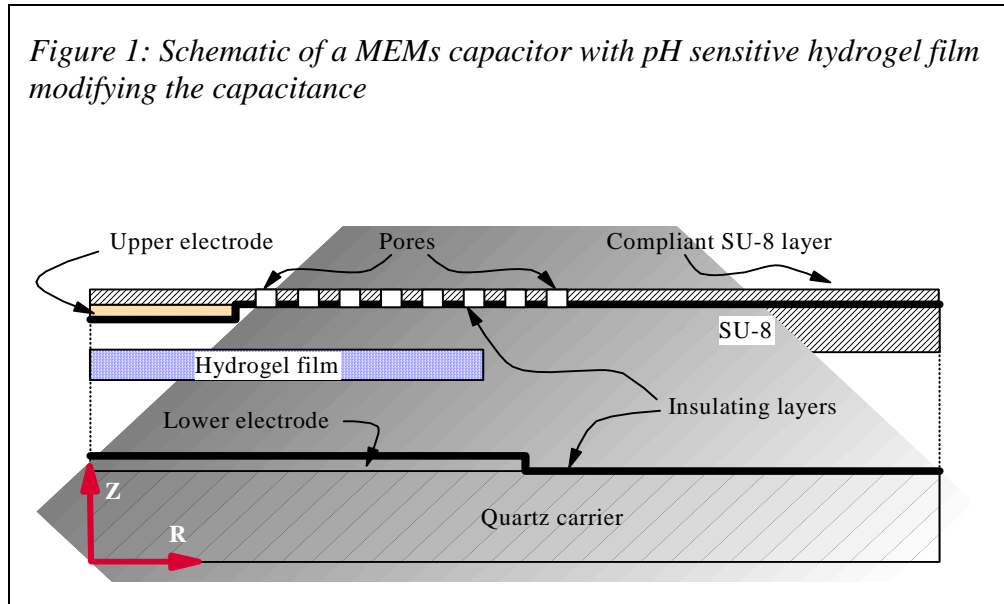
mercial products available, ranging from simple embedded thermocouples to complete “maturity systems”, which calculate the strength properties of concrete as a function of the collected time and temperature data. The prices of the embedded probes and loggers ranged from 40 to 100 dollars, and some came with external readers that could cost up to 2000 dollars. There were also wireless options available, which could transmit up to 8 inches in concrete without an external antenna. For humidity monitoring, there were less options available, and they all required drilling a hole into the already cast concrete. None of the products could be embedded into concrete during casting. There were no products available for in-situ monitoring of pH or chloride content in cured concrete. The available methods relied on taking a sample and analyzing it in a laboratory. The concrete monitoring device that ADC is currently developing will result in the capability for in-situ, wireless monitoring of also other parameters in addition to temperature.

### **3.2 Sensor Research and Development**

The main focus for this reporting period was on sensor design. We purchased several commercially available sensors for measuring humidity and temperature. Some of these were specifically designed for use in concrete, and gave us a chance to study the packaging schemes designed for this purpose. Other sensors, not currently used for concrete monitoring, were selected that were small in size or were electrically passive (capacitive or resistive). These are used for early learning about their operating principle and for integration into the RFID unit to evaluate its performance. The final goal is to produce miniature passive sensors that will be powered by the RFID signal. For the next reporting period, the main task will be designing and testing the RFID system. This will include programming an RFID chip so that it can read information from sensing elements and send the information to the transmitter.

The first sensors to be integrated to the RFID system will be the temperature and humidity sensors. The performance of the system will then be evaluated in concrete. Chloride and pH sensors are expected to be integrated to the system during phase II. ADC has a patented technology for a capacitive pH sensor, which is based on a pH sensitive hydrogel. ADC has already designed and tested a MEMs based hydrogel capacitor, where the capacitance changes with pH.

The critical requirement was to identify a photo-imagable material that could serve as the flexible electrode, survive millions of operating cycles, resist damage from the environment and be compatible with MEMS processing. SU-8 photoresist, first patented by IBM in 1989, was chosen for this purpose. SU-8 is a chemically amplified, epoxy-based, negative resist. Structures having exceptionally high aspect ratios and straight sidewalls are readily formed in thick films by contact-proximity or projection printing. Cured SU-8 is highly resistant to solvents, acids and bases and has excellent thermal stability, making it well suited for applications in which cured structures are a permanent part of the device. It is also highly elastic, with a yield strength of > 80 MPa, making it ideal for this application.



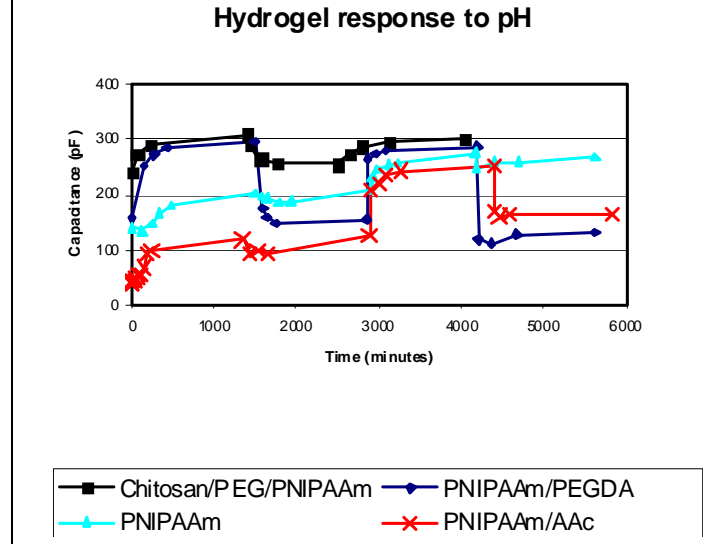
A schematic of the MEMS sensor is shown in Figure 1. The stress in the compliant layer is the limiting factor in the design; it is governed primarily by the material properties, the size and number of pores and the thickness of the hydrogel. A diameter of 2.5 mm was chosen for the cavity. Using results of FEM analysis, a thickness of 2.5  $\mu\text{m}$  was chosen for the SU-8 membrane. The device was designed to be built in three parts: a base consisting of an electrode permanently mounted on a quartz chip; a 2 $\mu\text{m}$ -thick hydrogel disk; and a perforated membrane of SU-8, with an electrode at its center and a ring of SU-8 beyond, forming the cavity into which the hydrogel would be placed. The hydrogel was spin-coated and die-cut by Prof. Chu at Cornell University using a device built by ADC. The other two parts of the device were built at the Cornell Nano-fabrication Facility and assembled at ADC.

Experiments were conducted with parallel plate capacitors in order to study the swelling response of various hydrogels to temperature, pH and salinity. As the hydrogel swells, a change in capacitance occurs due to change in its dielectric constant and deflection of electrode. Tests were conducted with four hydrogels: 85% poly(N-isopropylacrylamide) (PNIPAAm) copolymerized with 15% acrylic acid (AAc); 80% PNIPAAm copolymerized with 20% Poly(ethylene glycol dimethacrylate) (PEGDA); 70% Chitosan copolymerized with 20% PEG and 10% NIPAAm and a 100% NIPAAm. After an initial rinse in DI water, prototype sensors were immersed alternately in buffered solutions of pH 7 and 10 over several 24 hour periods. Capacitance was measured periodically. Temperature was held at  $21.5 \pm 0.5$   $^{\circ}\text{C}$  over the entire test. When a sensor is immersed in one of the buffered solutions, there is an immediate change in capacitance due to the permittivity of the surrounding fluid, about 80 times that of air. A gradual change follows as the hydrogel absorbs or desorbs water over a period of about 24 hours. Two phenomena are at work simultaneously: (1) the permittivity of the hydrogel increases as water is absorbed, increasing the capacitance and (2) absorption of water causes a swelling that moves the electrodes further apart, decreasing the capacitance. If water is being desorbed the effects are the opposite of those described. The first of these effects was shown to be more pronounced, as seen in Figure 2. When first placed in the buffered solution at pH 7 each sensor rapidly absorbed water, increasing its

capacitance. All required more than 24 hours to stabilize, most likely due to the large dimensions, with Chitosan/PEG/PNIPAAm and PNIPAAm/PEGDA samples responding more rapidly. These two also provided the most sensitive and repeatable responses. The capacitance of the Chitosan sensor changed by about 15% while the PNIPAAm/PEGDA sample changed nearly 50%. For use with passive RF devices, where the change in capacitance is used to change the resonance frequency of the antenna network, either material would require additional capacitance in parallel to reduce the magnitude of the change.

Further testing is still needed to choose the optimal pH sensing film and evaluate its response to pH and temperature. Chloride sensor can be built with the same technology and using a chloride sensing thin film instead of the pH sensing film.

*Figure 2: pH response of four different hydrogels cycled between pH 7 and 10*



### **3.3 Consultation with the Concrete Lab at Cornell University**

Alex Deyhim and Tia Korhonen visited the George Winter laboratory at Cornell University to meet with lab manager Tim Bonds and discuss the project. The topics discussed included the currently available measuring methods for temperature and humidity and their limitations. Tim Bonds noted that the sensor that is currently being developed will also be very useful for making measurements of soil conditions. We also discussed the possibility and costs of testing the sensors in an actual concrete structure during the phase II evaluation of the sensor performance. Tim Bonds suggested that the most cost effective way would be if they were able to place the test sensors in concrete structures to be built for one of their existing projects. Parameters to be tested will include performance and range in concrete of the radiofrequency communications system, as well as adequateness of packaging and the performance of the actual sensors elements.

### **3.4 Discussions and Presentation by Professor Hover**

Professor Ken Hover from Cornell University visited ADC two times to discuss the project. According to him, people are increasingly using temperature sensors with ibutton packaging schemes to monitor state of concrete during curing. These are very small and cheap enough that the cost is not a limitation and several of them can be embedded in different locations of the structure during pouring of the concrete. There are currently no commercially available, low-cost methods for measuring humidity from embedded sensors, and the options that are available are only meant for measuring humidity for the first week or so during curing.

Professor Hover suggested that we might consider developing and commercializing two different products, one for monitoring the temperature and moisture during curing of the concrete in the

first few weeks, and the other one for long-term monitoring the health of concrete during service life. During curing of concrete, temperature and moisture gradients can develop, either between parts that were cast at a different time, or between the surface and the bulk of the material. Since temperature controls the rate of hydration and strength development, and because adequate moisture content during curing controls the extent of hydration and the strength of the final structure, embedded monitors in several locations are important, to be able to track the mechanical properties at different locations in the concrete structure. For monitoring the strength of concrete during service, and the rate of deterioration, parameters such as chloride concentration and pH become important. Corrosion of the reinforcing steel bars is driven by a combination of moisture, temperature, and chloride concentration. In concrete that has not been contaminated by deicing salts or sea salt, the reinforcing bars are protected against corrosion by the high pH (up to 13) of Portland cement concrete. If the chloride ions penetrate the concrete and lower the pH, the reinforcing bars can corrode. The reinforcing bars are placed just beneath the surface of the concrete, which is the first part of the structure to be permeated by chlorides and other diffusing species from the outside environment. Therefore, the chloride sensors and other ion-specific sensors should be placed within the first few inches of the structure.

Professor Hover also gave a 2-hour presentation on concrete for the key technical people at ADC. The main theme of the presentation was to give us an appreciation of how complex a material concrete is. The physical, chemical and mechanical properties of concrete depend on several interconnected parameters, including the initial properties of the aggregates and the cement, as well as the conditions during curing. Hardness of the concrete develops during a sequence of hydration reactions that cause the CaO and SiO<sub>2</sub> in the cement paste to form crystalline compounds. The size and density of the crystals as well as the distributions of pores between the crystals all contribute to the final strength of the cured concrete. Porosity also determines concrete permeability for water, oxygen, chlorides, carbon dioxide, and other substances that can degrade concrete long-term performance. It is very useful to be able to record the temperature-time history during curing of concrete in order to estimate its structural strength. The maturity method estimates of the degree to which the Portland cement has been hydrated from a non-linear rate relationship between concrete temperature and the amount of cement hydration. The hydration is controlled by the temperature and moisture content during curing, and if the concrete is not handled properly, then it will not develop the structural strength that it is designed for.

#### ***4. Upcoming Tasks for Progress Report #2***

##### ***4.1 Development of an RFID communications system***

Develop an RFID communications system that is able to read the information from the sensors. The system will consist of a programmable RFID chip, a transmitter, and the sensors. When activated by a signal from the transmitter, the RFID chip will read the information from the sensors and relay this information back to the transmitter.

#### **4.2 Start integrating the sensors into the RFID system**

The first sensors to be integrated will be the temperature and humidity monitoring sensors. We have purchased several temperature and humidity sensors, which will be integrated to the RFID system, and the system will then be tested in different temperatures and moisture contents

#### **4.3 Test the performance of the measuring system**

Test both the performance of the sensors in measuring the environmental condition (at this point, temperature and humidity) and the proper functioning of the RFID communication system